NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

VOLUME 49
NUMBER 2
FEBRUARY 1965

U. S. DEPARTMENT OF COMMERCE

U.S. Department of Commerce — John T. Connor, Secretary

National Bureau of Standards — A. V. Astin, Director

NATIONAL BUREAU OF STANDARDS

Technical News

BULLETIN

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. Subscription price: domestic, $1.50 a year; 75 cents additional for foreign mailing; single copy, 15 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (June 19, 1961).

Library of Congress Catalog Card Number: 25-26527

February 1965 — Issued Monthly — Vol. 49, No. 2

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COVER: Dr. Reatha C. King prepares the analysis train used to obtain an improved value for the heat of formation of the refractory compound aluminum carbide (p. 26). Results of this work give information about the stability of other carbides used as refractory materials in industrial processes. Combustion of the aluminum carbide sample takes place in the combustion bomb (bottom, center).

One-Pan Balance Speeds Weighing of Large Loads

Instrument devised at the NBS Institute for Basic Standards

A rugged, fast-operating balance has recently been devised for weighing loads up to 50 pounds. Evaluation tests of a commercially constructed prototype demonstrate that the instrument [1] has a high degree of precision, even under undesirable weighing conditions. In addition, the simplicity of its configuration, the rapidity with which precise readings may be made, and the elimination of tedious calculations required by other techniques make the new balance particularly suitable for use by State weights and measures officials, as well as by industrial laboratories where weighings of large loads are regularly carried out.

The calibration of weight standards at the 50-lb level, either a single weight or a set of weights totaling 50 lb, requires precision in the measurement equipment to at least a part in 500,000. As a rule, a conventional two-pan three-knife balance, enclosed in a wood and glass cabinet to inhibit atmospheric effects, is employed for this work.

In this method, however, the delicate remotely controlled manipulations necessary to complete a calibration are time-consuming. A series of computations must also be carried out to determine the mass corrections required for standard weights. A study was therefore undertaken to devise a balance that would accommodate the bulk of large weights, would accomplish the necessary manipulations, would eliminate the required computations, and would achieve precisions to a part in 500,000 under controlled laboratory conditions.

In the study, the possibility of adapting the one-pan two-knife type of balance now used for weighings of loads varying from a small fraction of a pound up to about 6 lb was explored. Attached to one end of the balance beam of this widely used device is a counterweight which balances a loaded suspension coupled to the opposite end of the beam by means of an end-knife assembly. The beam oscillates about a second knife centered between the end knife and the center of gravity of the counterweight. Built into the instrument is a group of small tare weights which are manipulated to vary the load on the suspension. These weights are of such magnitude that their summed moment about the center knife precisely balances the moment of the counterweight.

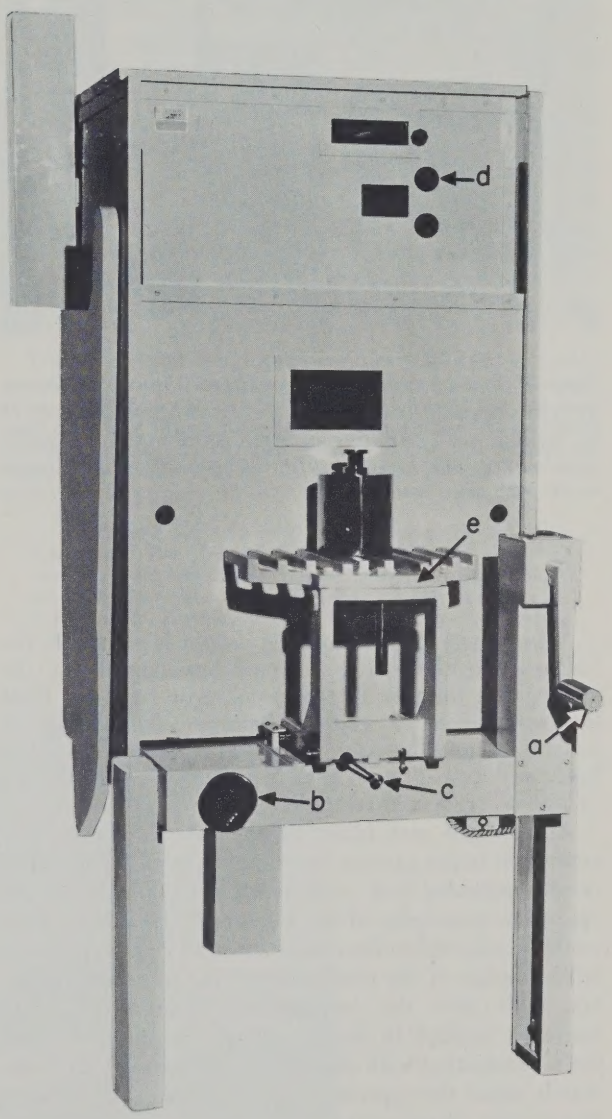

Figure 1. *High-precision balance. Control levers* **a** *and* **b** *are for manipulation of a weight-transfer mechanism inside the cabinet,* **c** *is a balance-pan-release control,* **d** *is a knob for adjustment of a graduated scale, and* **e** *is a mobile device for transporting heavy loads inside the cabinet.*

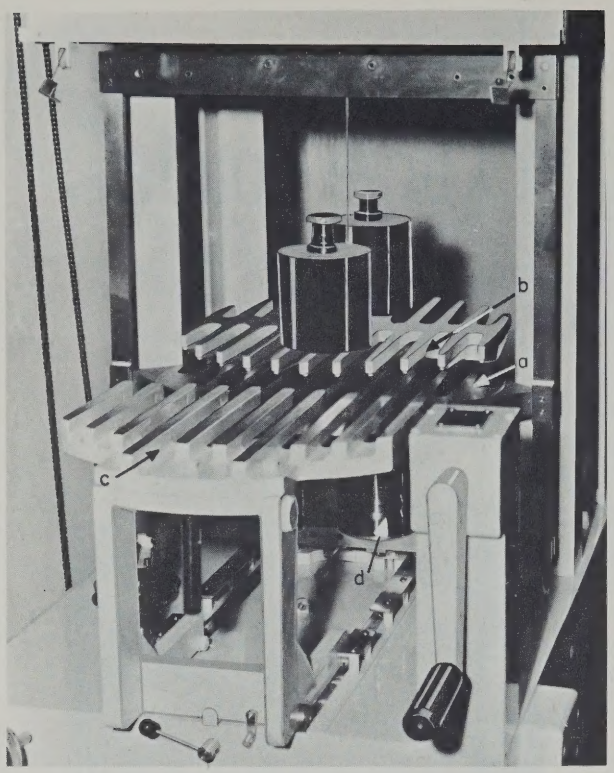

Figure 2. *Lower chamber of high-precision balance. The balance pan* **a** *is slotted so that similar slots on turntable* **b** *can be fitted into the balance pan to accomplish weight transfer. Similar slots on the "truck"* **c** *permit transfer of a weight from the truck to the table. One of a pair of tare weights* **d** *is shown on the pan-hanger assembly.*

When a test load of nominal weight is placed on the balance pan, the system becomes unbalanced and the appropriate number of tare weights is removed from the suspension to restore balance. The mass of weights removed approximately equals that of the test load, and any difference is indicated on a graduated "readout" scale calibrated directly in terms of mass.

An analysis was made of the inherent errors encountered in the precise weighing of large loads. The results indicated that, with suitable modifications, the operation principles of the one-pan two-knife balance could be adapted to the construction of a 50-lb balance. In the design of the new balance,[2] the tare weights are not built into the instrument. Instead, they are manually applied to the pan-hanger assembly so that their summation with the unknown load will approximately equal the capacity of the balance. An unusual feature also incorporated in the design is a mechanism for remotely transferring a standard weight or a test load to and from a storage position inside the instrument to facilitate the substitution weighing technique[3] used in NBS mass calibrations.

Instrument Design

The instrument constructed according to this design is shown in figure 1. It consists essentially of a $42 \times 25 \times 26$ in. cabinet having two chambers: an upper one which holds a 50-lb counterweight and a beam assembly, and a lower one containing a pan-hanger assembly and a slotted turntable to accomplish weight transfer. When the lower front panel of the cabinet is raised, a top-slotted "truck" (*e*, p. 23) mounted on the outside and used to transport a weight into the cabinet, can be easily moved along the ball-bearing slides in the floor of the lower chamber.

Both vertical and horizontal motions of the turntable are accomplished with control levers on the front of the cabinet (*a* and *b*, fig. 1). A lever (*c*, fig. 1) that controls a pan-release mechanism and a knob (*d*, fig. 1) for adjustment of the instrument's graduated scale are also on the front of the cabinet.

In addition to the counterweight-beam assembly in the upper chamber, suitable damping and arrestment devices were installed to control beam and pan-hanger motion. A conventional optical projection system, also in the upper chamber, permits easy reading of the graduated scale.

Weighing Procedure

In the first weight-loading step, the lower panel is raised (fig. 2) and a standard weight and a test load are transferred successively from the truck (*c*, fig. 2) by manipulation of the slotted turntable (*b*, fig. 2) through the slots of the truck. The turntable is then moved to a raised position above the balance pan so that it can be rotated to present either of the two weights to the pan (*a*, fig. 2) below. To carry out a substitution weighing, readings are made first with a standard weight on the balance pan, and second, with a test load on the pan. In this procedure, the required tare weights (*d*, fig. 2, indicates one of a pair of weights) are stacked symmetrically on the pan-hanger assembly.

Results

Numerous substitution weighings of standard weights have thus far been accomplished with this instrument, and precisions to a part in 10^6 have been routinely obtained. Models constructed along the lines of the NBS prototype would provide a practical means for making weight comparisons in the 10- to 50-lb range.

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[1] *For further technical details, see* A direct-reading two-knife 50-lb balance of high precision suitable for State weights and measures laboratories, *by H. E. Almer, H. A. Bowman, M. W. Jensen, L. B. Macurdy, H. S. Peiser, and B. Wasko, J. Res. NBS* **68C** *(Eng. and Instr.), 141 (July–Sept. 1964).*

[2] *Designed for NBS by B. Wasko, Voland Corp., New Rochelle, N.Y.*

[3] *For details on substitution weighing, see* National Bureau of Standards Precision Measurement and Calibration, *NBS Handb. 77, Vol. III (Optics, Metrology, and Radiation), p. 655, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402,* $7.00.

performance of "SLOW-FLOW" METERS

The NBS Institute for Applied Technology recently investigated the performance of small "slow-flow" meters which could be used to measure fuel piped to home oil burners from a remote supply source. Four meters representative of those on the market were tested and gave satisfactory results. Such meters may have wide utility in areas where a centralized fuel-oil storage and distribution system is economically feasible, as, for example, in new housing developments and in trailer parks.

A number of State officials had approved all but one of the components required for large-scale distribution of fuel oil from a central facility. The exception was the device intended to measure the amount of fuel consumed. In order to meet State weights and measures requirements, this component had to be accurate while measuring extremely slow flows. The so-called slow-flow meter was designed for such measurements, but its accuracy in measuring liquid fuels was not known to weights and measures officials. At the request of State officials, Donald R. Mackay of the NBS Office of Weights and Measures therefore undertook the present study to determine this accuracy, and to devise a simple test method for use by State officials in evaluating slow-flow meters.[1]

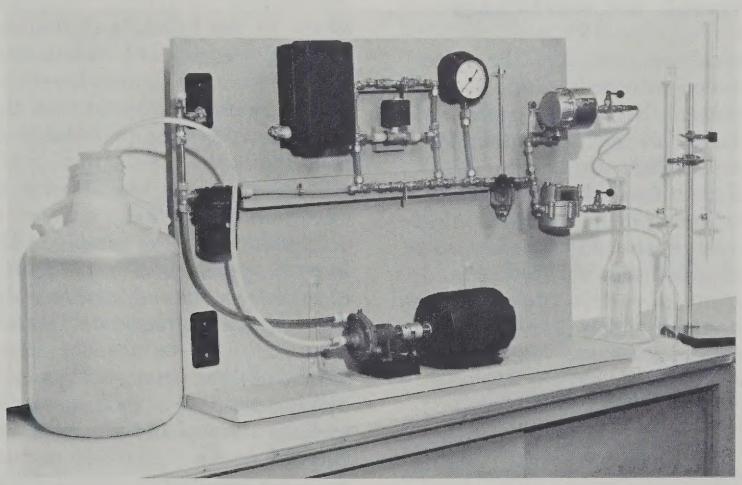
Meters submitted by four manufacturers were accepted for test. All were piston-type, positive-displacement meters, three with single, double-acting pistons, and one with four pistons.

A test panel was designed and built to accommodate the control mechanism necessary to determine meter accuracy. The parts fitted to the panel included a polyethylene carboy to provide a 5-gal supply of test fluid to a piping system mounted on the panel, a centrifugal pump to provide flow under pressure, valves to control line pressure and flow rates, and connectors for attaching two meters at a time to the system. Glass flask standards ranging in size from 1 gill to 1 gal were used to determine the actual amount of liquid each meter delivered during a test. This amount was then compared with the amount shown as delivered on the meter register to determine meter accuracy.

Multiple tests were conducted on each meter at constant flow rates varying from 0.05 to 3.6 gal/hr. In one series of tests, varsol was employed for safety reasons to simulate the type of fuel that is used for space heaters in mobile homes, garages, and utility buildings. In another series, fuel oil used by gun-type burners such as those installed in residences and large buildings was employed. In the varsol tests, the *average* overregistration of the meters was only 0.36 percent, and in the fuel tests it was even less—0.06 percent.

Tests in which the supply of fuel was intermittently turned on and off were also conducted, as well as tests at low temperatures to simulate actual operational conditions. The results obtained showed that neither of these variables substantially affected meter performance.

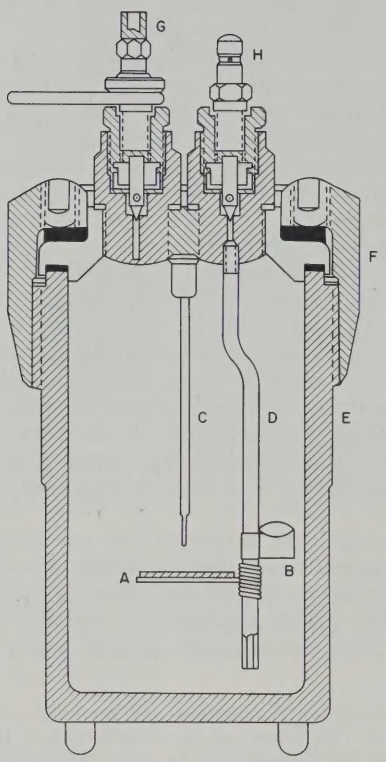
[1] *For details on test method and procedure, see* NBS Tech. Note 196, Report of the investigation of slow-flow meters for fuel oil distribution systems, *by D. R. Mackay, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (20 cents).*



Equipment used to test "slow-flow" meters. To the left of the panel is a polyethylene carboy which provides a 5-gal supply of test fluid to the piping system mounted on the panel. On the right side of the panel are mounted two meters to be tested.

Heat of Formation of ALUMINUM CARBIDE

Investigation by
R. C. King and G. T. Armstrong
of the NBS Institute
for Basic Standards



Cross-sectional view of combustion bomb. **A**, *platinum-iridium support loop, with alumina disk, for aluminum carbide sample;* **B**, *platinum crucible for combustion of benzoic acid;* **C**, *electrode to ignite the sample;* **D**, *inlet tube for gases;* **E**, *bomb base;* **F**, *screw cap;* **G**, *outlet valve;* **H**, *inlet valve for gases.*

high thermal stability

Thermochemical properties of the refractory compounds have proved extremely difficult to determine accurately. A recent NBS investigation has resulted in an improved value for the heat of formation of the refractory compound aluminum carbide.[1]

This compound, although it has a high thermal stability, is too chemically reactive to be used practically as a refractory material. However, a determination of the heat of formation of aluminum carbide gives information about the stability of certain other carbides which are nearly inert and are widely used as refractory materials in industrial processes.

Refractory compounds, having a high temperature stability, extreme hardness, and extreme chemical inertness, are used as furnace linings, especially for roasting and smelting in metal-refining processes. Knowledge of the thermochemical properties of these materials is needed to determine the temperature range of stability, the conditions of formation, and the heat balances in processes in which they take part. Such data are also essential to some calculations in basic research problems.

oxygen bomb calorimetry

In the present study, the heat-of-formation data were obtained by means of oxygen bomb calorimetry.[2] The combustion bomb, made of corrosion-resistant alloy steel and equipped with a platinum-iridium loop to support the sample, was placed into a Dickinson-type calorimeter. Purified oxygen under pressure was added to the bomb, and the sample was ignited electrically by means of a nickel-chromium fuse wire. The heat of reaction of aluminum carbide with oxygen was measured and combined with the heat of formation of the products, aluminum oxide and carbon dioxide, to yield the heat of formation of aluminum carbide.

Particular care was taken to eliminate errors due to uncertainties in the analysis[3] and in the incomplete combustion of the sample. This work also differed from that of earlier investigators in (1) the exclusion of carbonaceous combustion aids from the reaction, (2) the absence of elemental aluminum as a combustion promoter, (3) the absence of water in the combustion bomb, (4) the determination of the amount of reaction by measurement of the mass of CO_2 formed, and (5) the identification of the crystal forms of the solid combustion product. Failure to control these factors apparently contributed to considerable scatter in the data of earlier studies of the thermochemical properties of aluminum carbide.

(*continued opposite*)

NBS Low-Frequency Station WWVB to Broadcast International Unit of Time

Following the adoption by the 12th General Conference of Weights and Measures of a supplementary definition of the second based on the invariant transition of the cesium atom, the Bureau has begun broadcasting the new international unit of time from its Fort Collins, Colo., low-frequency station WWVB, effective January 1, 1965.

This action follows the request by the U.S. Study Group VII, of the International Radio Consultative Committee (CCIR), for a study to determine the most useful systems for broadcasting the various kinds of time signals which find use in civil and scientific affairs.

A recent users' survey conducted by the National Bureau of Standards reveals that at least a third of the Nation's electronics establishments not only use the NBS frequency and time broadcasts as the basis for precise measurements in their operations, but that they expressed a wish for exact atomic time and frequency by direct broadcast. These users include the largest governmental and industrial laboratories, precision instrument manufacturers, space hardware manufacturers, satellite launch facilities, and missile-tracking facilities.

The standard carrier broadcasts from WWVB will be maintained without offset with respect to the United States Frequency Standard (USFS) within a tolerance limit of $\pm 2 \times 10^{-11}$.

Time pulses are produced by amplitude modulation of the carrier. Each pulse, initiated at the same relative phase of a cycle of the carrier, is repeated at intervals of 60 kilocycles of the 60 kHz carrier—or once each second (the international or atomic second). Thus the new international unit of time is directly available to listeners for the first time.

The phase of these periodic time pulses relative to the carrier phase will be shifted by a step adjustment of 200 msec, or exactly 12,000 whole cycles of the carrier, about once every 3 months or so in order to remain within approximately 100 msec of the UT2 scale which is broadcast by the other NBS stations. Adjustments will always be carried out at 0000 UT on the first of each month. The first such adjustment was made on January 1.

Keyed Morse Code announcements, which provide the time difference, UT2 minus time of pulse, will be broadcast in the same manner as for WWV and WWVH. The difference in milliseconds will be broadcast during the identification period on the hour and every 20 min thereafter in International Morse Code. The code, UT2 AD (for add) or UT2 SU (for subtract) is followed by a three-digit number. Added to or subtracted from the time indicated by the time pulse, the correction will give the UT2 scale. The corrections will be revised daily, the new value appearing for the first time during the day after 0000 UT, and will be repeated for the ensuing 24-hr period. NBS obtains its UT2 information weekly from the U.S. Naval Observatory in accordance with the close cooperation maintained by the two agencies.

Advance announcements of step adjustments will be made in the *Federal Register* so that listeners may be adequately prepared.

Aluminum Carbide, cont.

crystalline forms

In the NBS experiments, it was found that the completeness of combustion of the aluminum carbide sample was related to the heat capacity of the sample container in the combustion bomb. By use of small alumina disks as supports for the sample on the platinum-iridium loop, combustion of more than 99.5 percent of the aluminum carbide was achieved. The value for the heat of formation of aluminum carbide was determined to be -208 kJ/mole (-49.7 kcal/mole) with an overall uncertainty of ± 5 kJ/mole (± 1.2 kcal/mole).

An interesting result of the study was the finding that 45 to 75 percent of the solid combustion product was the delta crystalline form of aluminum oxide (alumina) which condensed on the walls of the bomb as a fine powder. The rest of the solid product, a massive lump remaining in the combustion zone, was the alpha crystalline form of alumina. The conditions under which delta alumina has been reported to be formed readily (presence of carbon and nitrogen, and rapid condensation and cooling from the gas phase) all were present in these experiments and may explain the formation of such a large amount of this less well-known form of alumina.

In previous investigations, little if any mention was made of the crystalline forms of alumina in the combustion product. The present work shows that for accurate heat-of-formation data from calorimetric studies of refractory compounds, giving solid combustion products, it is important to identify the various crystalline forms in the combustion product and to correct the heat measurements for their formation. Information gained in these experiments, regarding delta-alumina formation, may be of value in describing aluminum reactions in the environment of a rocket motor, which is chemically similar to the environment in which delta alumina is formed in bomb calorimetric experiments.

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[1] The heat of combustion and heat of formation of aluminum carbide, *by R. C. King and G. T. Armstrong, J. Res. NBS* **68A** *(Phys. and Chem.), 661 (1964).*

[2] Precise measurement of heat of combustion with a bomb calorimeter, *by R. S. Jessup, NBS Mono. 7 (1960).*

[3] *The aluminum carbide sample for this study was supplied by the Aluminum Company of America Research Laboratories. Quantitative chemical analysis showed in weight percent:* Al_4C_3, 94.8; Free Al, 1.0; Free C, 1.0; AlN, 1.3; Al_2O_3, 2.2; Fe, 0.06.

ALUMINUM CORROSION PROTECTION

pH is a controlling factor

Investigation by
W. J. Schwerdtfeger of the
NBS Institute for Materials Research

Externally applied currents have long been used to protect iron and steel from corrosion in underground and aqueous exposures. When the same technique has been used on aluminum in corrosive environments, however, contradictory results have been obtained. To resolve this conflict, recent experiments have measured the corrosion rates of specimens of an aluminum alloy under a variety of exposure conditions and with external current applied to some of the specimens.

Data thus derived showed that the electric technique can block aluminum corrosion in some acidic environments, but that it may accelerate aluminum corrosion in alkaline environments.[1] These findings indicate the possibility of electrically protecting aluminum underground structures, such as pipelines and storage tanks, when the *p*H of the environment is taken into consideration.

The reaction of metal to a corrosive environment is essentially an electrochemical phenomenon. Differences in electric potential develop at the surface of the metal, causing electric currents to flow through the electrolyte. Thus metal is lost from anodic areas and hydrogen is evolved at cathodic areas on the metal surface.

In the electric technique used for metal protection (known as cathodic protection), the current between the specimen and an auxiliary anode and the potentials between the specimen and a standard reference electrode are measured as increments of current are applied. A characteristic change in slope or "break" in the curve derived for this current-potential relationship denotes the current that will block the corrosive reaction on the metal surface.

In the present study, the effects of applying the method to the specimens in salt water and in acidic and alkaline salt-water solutions were determined. The potentials of aluminum alloy specimens in air-free soils covering the *p*H range peculiar to soils were also measured in an effort to select a protective potential value for these alloys.

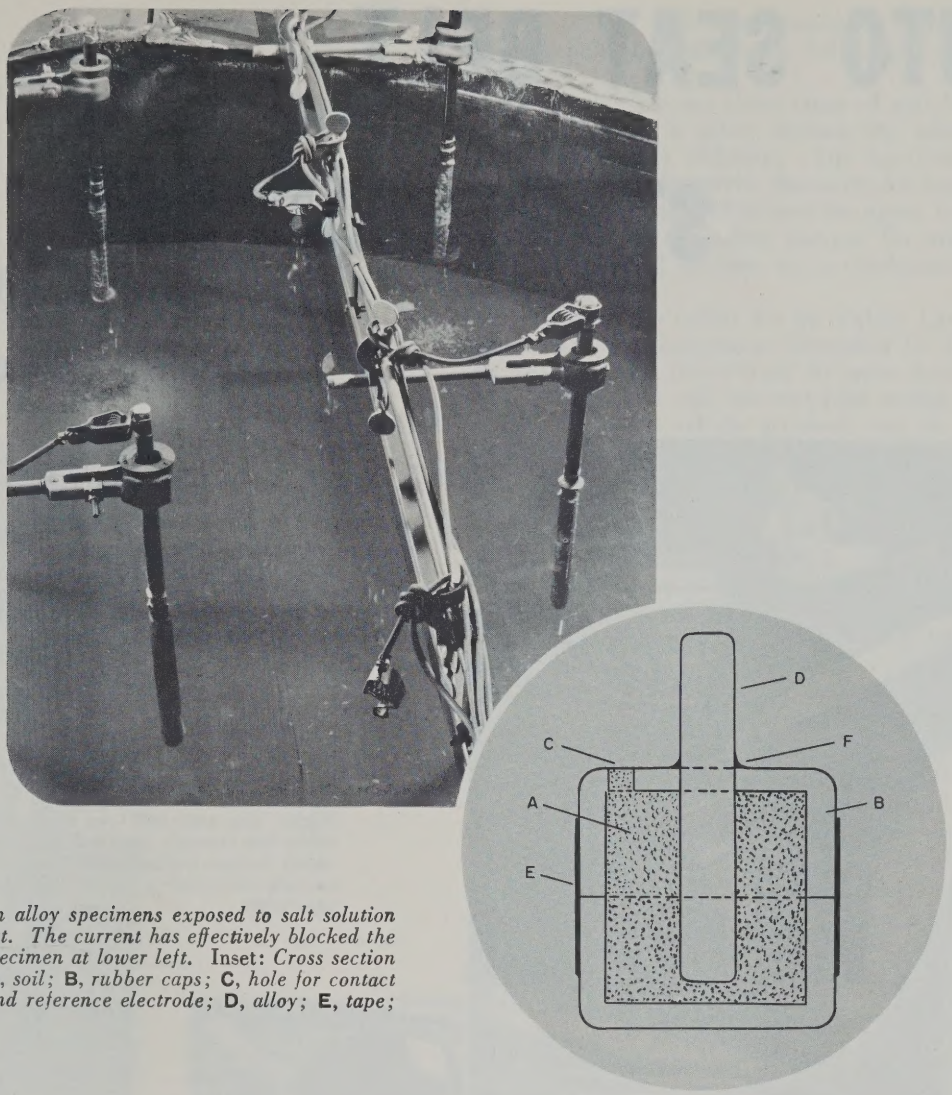
Experimental results indicated that the application of electric currents to aluminum increases the alkaline concentration of the environment. Apparently, in low-*p*H (acidic) environments this concentration has a neutralizing effect; however, the increased alkaline concentration may be deleterious in high-*p*H environments. These results are attributed to the amphoteric nature of aluminum, which—unlike many other metals—can be highly reactive to both types of environment. Contrary to other findings, the results also indicated that use of a specific potential value for the protection of aluminum in all underground exposures is not possible.

Salt-Water Experiments. In the salt-water experiments, three "cathode" specimens (to which current was continuously applied) and three controls (no applied current) were suspended vertically in a large wooden vat and exposed to city water to which 3 percent by weight of sodium chloride had been added. The different currents applied to each of the cathodes were based (1) on a selected potential of −1.5 V; (2) on periodic potential measurements (−0.794 to −0.842 V) of the controls; and (3) on the potential (−1.0 V) of a coupled zinc electrode which served as a sacrificial anode for the third cathode. All potentials were measured with reference to a saturated calomel electrode.

At the end of approximately 6 months' exposure, the specimens were removed from the vat and their weight losses were measured. These measurements showed that the cathode adjusted to −1.5 V suffered severe corrosion and that the potentials at which the other two cathodes had been held provided cathodic protection.

Acidic and Alkaline Solutions. In the second series of experiments, two sets of specimens, each consisting of a cathode and a control, were placed in two jars containing a salt solution adjusted to *p*H 4 by the addition of acetic acid. Two similar sets of specimens were placed in salt solutions adjusted to *p*H 10 by the addition of sodium carbonate. Weekly electrical measurements were made on all the specimens over a 6-month exposure period.

Potential-current curves obtained from the controls in the acidic salt solution served to furnish protective criteria for one of the cathodes, and also to permit calculation of the normal rates of corrosion from time to time. It was thus possible to compare the magnitudes of the currents applied to the cathodes with the concurrent corrosion currents associated with the controls.



Four aluminum alloy specimens exposed to salt solution in a wooden vat. The current has effectively blocked the corrosion of specimen at lower left. Inset: *Cross section of soil cell.* **A**, *soil;* **B**, *rubber caps;* **C**, *hole for contact between soil and reference electrode;* **D**, *alloy;* **E**, *tape;* **F**, *sealer.*

One cathode in the acidic solution was controlled within the potential range −0.775 to −0.935 V (derived from the controls' potential-current curves), and the other cathode was held at a potential between −1.00 and −1.05 V. Measured weight losses at the end of the exposure period showed that the cathodic protection was achieved on both, but to a slightly better degree on the latter.

In the alkaline salt solution, one cathode was controlled within the potential range −1.30 to −1.49 V (as indicated by the controls' potential-current curves) and the other to within −1.45 to −1.55 V. From measured weight losses at the end of exposure it was found that both cathodes had much higher corrosion rates than the controls, indicating that the application of cathodic currents in an alkaline environment accelerates corrosion. The corrosion rates of these controls, as revealed from potential-current curves, diminished greatly toward the end of the exposure period.

Soil Environments. In the third series of experiments, aluminum alloy rods were exposed to 21 different soils ranging in *p*H from 3.4 to 10 for a 6-month period under air-free conditions. Weekly potential measurements disclosed no consistent relationhsip between soil *p*H and alloy potential. The specimen in the most alkaline soil (*p*H 10) had the most electronegative potential (−1.4 V), while the most electropositive potential (−0.68 V) was measured in a soil of *p*H 8.8.

The range of potentials (0.7 V) measured in the laboratory, together with experience in the field, indicates that a single value as a protective potential for aluminum alloys in all underground environments cannot be specified. Instead, the combined data suggest that the existing corrosion potential of an alloy be shifted about 0.1 V in an electronegative direction to achieve cathodic protection, and that caution be exercised in extremely alkaline soils.

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[1] *For details, see* Effects of cathodic currents on the corrosion of an aluminum alloy, *by W. J. Schwerdtfeger, J. Res. NBS* **68C** *(Eng. & Instr.), No. 4 (Oct.–Dec. 1964).*

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AUTO SEAT BELT STANDARDS SET

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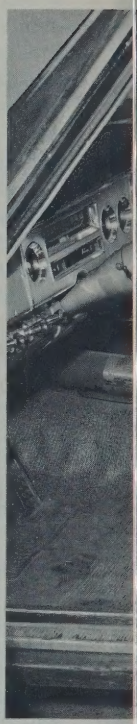
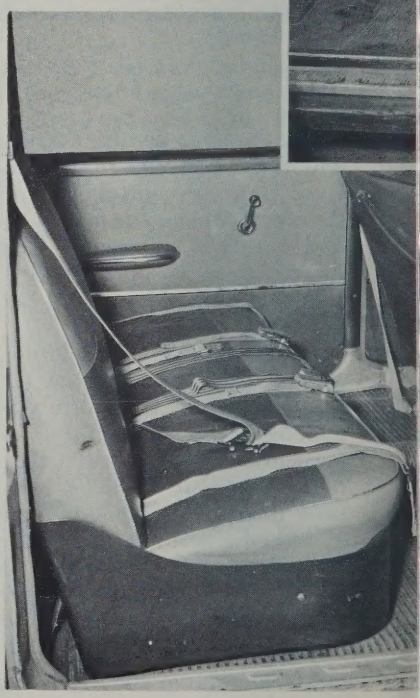
Left: *Mrs. Margaret Turner wears the safety harness. Lap and shoulder units buckle separately.* Below: *James E. Walker wears the one-buckle adult safety harness. This belt is an integral lap and shoulder unit.*



Right: *Lap seat belt, one type of automobile safety belt recently standardized.* Below: *Adult safety harness utilizing both a lap seat belt and an over-the-shoulder belt. The belts buckle separately.*



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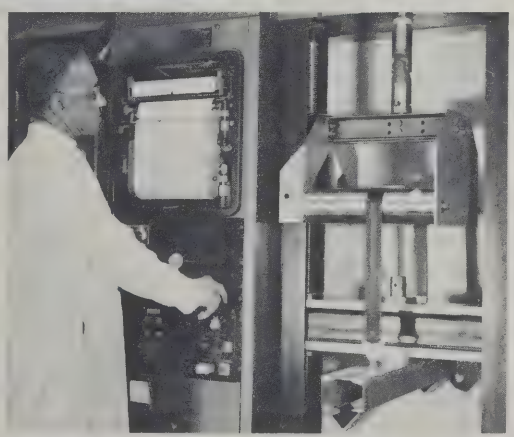

Russell J. Capott adusts the testing machine used to determine the strength of a completely assembled seat belt

The U.S. Department of Commerce has recently published standards for motor vehicle seat belts.

The standards cover three types of seat belt assemblies: a lap belt, a safety harness for adults, and a safety harness for children. The lap belt provides restraint only on the pelvis; the safety harness provides restraint on both the pelvis and the upper torso. The standards permit the safety harness for adults to be either an integral harness or a combination of lap and shoulder belts.

The action was called for by Public Law 88–201, approved by the Congress on December 13, 1963. The law required the Department to issue standards "to provide the public with safe seat belts so that passenger injuries in motor vehicle accidents can be kept to a minimum." The standards were published in the Federal Register, Friday, December 11, 1964. The manufacture, sale, or transportation in interstate commerce of seat belts which do not meet the standards will be unlawful beginning December 11, 1965.

The technical work for the seat belt standards was done by the National Bureau of Standards in cooperation with the Society of Automotive Engineers and an ad hoc committee of representatives of interested Federal agencies. The standard is essentially the same as Society of Automotive Engineers Standard J4b.

Prior to June 1, 1965, the Department of Commerce, in consultation with the Society of Automotive Engineers, the industry, Government experts, and interested consumers, intends to add additional requirements to the standard. The proposed additions will be published in the Federal Register for public comment about April 1, 1965. In the future it is expected that a dynamic test simulating collision will be included in the seat belt specifications which at present are based on a series of static tests.

The standard requires that the completely assembled lap belt (including buckles, straps, adjustment and attachment hardware, and retractors for webbing storage) be able to withstand a force of 5,000 lb. (2,500 for each component). For the adult safety harness, the structural components of the pelvic restraint must withstand 2,500 lb. and those of the upper torso restraint, 1,500 lb. Components common to the pelvic and upper torso restraints must withstand a force of 3,000 lb. The complete assembly of the child safety harness must withstand a force of 2,000 lb.

Requirements for the webbing material include retention of strength after abrasion, prolonged exposure to light, or exposure to the action of microorganisms, such as those causing mildew. The fabric must also be resistant to stains, to color changes caused by exposure to light, and to transfer of color upon rubbing or when wet. The hardware used must be corrosion resistant and free from burrs or sharp edges which might catch on clothing. Plastic components must not be subject to any warp or deformation within the range of normal operating temperature.

VLF SIGNALS:
Sunrise & Sunset Fading

Studies made by
D. D. Crombie of the NBS
Central Radio Propagation Laboratory
at Boulder, Colo., with ARPA support

It has long been known that, during sunrise, very-low-frequency (VLF) signals received over a long path show periodic variations in amplitude. At sunset, similar though smaller variations occur. Observations in recent years of the relative phase delay of such signals show that the amplitude variations are accompanied by phase variations.

Data on the periodic changes in amplitude and phase of VLF signals received at sunrise and sunset over long paths led Mr. Crombie to conclude that the variations are due to multi-mode propagation in the nighttime portion of the path.[1]

The original explanation was that during the day the ionosphere behaves like a metallic reflector having a pseudo-Brewster angle (angle of incidence for which the reflected signal is weakest) near 90° (grazing incidence), while at night it behaves like a dielectric with the pseudo-Brewster angle near 30°. During sunset or sunrise this angle changes from 30 to 90° and at some instant passes through a value equal to the angle of incidence of the wave. The signal level then decreases at this time because of the reduced reflection coefficient. This drop in amplitude of the received signal would then be repeated each time the sunrise (SR) or sunset (SS) line passed each "point" of reflection on a multihop path. This explanation now seems unlikely to be valid since calculations of the reflection coefficient of the ionosphere for VLF waves show that the decrease in reflection coefficient at the pseudo-Brewster minimum is small.

Recent explanation of phase variations. More recently an attempt was made to explain the periodic phase variations observed during sunrise and sunset at Neuchatel, Switzerland, of the 18-kHz signals from station NBA in the Canal Zone. This explanation, which is again based on a single-ray geometrical-optics approach, is that the five "steps" in the sunrise-phase variation are due to the SR line advancing over each of the five reflection points of the five-hop path. But this explanation does not attempt to account for the amplitude minima which accompany the phase "steps."

Explanations of the sunset and sunrise phenomena which are based on a ray-optics model, such as those mentioned previously, and which invoke only one ray seem unlikely to be able to account correctly for the observations on long paths for two reasons: First, it is necessary to include many rays in explaining VLF propagation over great distances, whereas only a few low-order modes are necessary. Second, it is necessary to include corrections for diffraction when using ray-optic models on a curved earth. Mr. Crombie has now accounted for fading on long VLF paths as due to interference between the two lowest order modes propagating in the nighttime portion of the earth-ionosphere waveguide.

Observations of the diurnal phase and amplitude variations of the signals from several distant VLF transmitters have been made at the Central Radio Propagation Laboratory, and at the Battelle Institute, Frankfurt, Germany, for more than a year. The two paths that are sufficiently long and correctly oriented to show well-developed sunrise and sunset fading with fairly good signal-to-noise ratio are NPM (Hawaii to Boulder, Colo.), distance 5400 km, using 19.8 kHz; and NBA (Canal Zone to Frankfurt, Germany), 9099 km, using 18.0 kHz. Typical diurnal amplitude and phase records for each of the two paths are shown in the graph. These curves represent most of the features of interest.

Major features of fading. One year's observation of the periodic variations of the phase and amplitude of these VLF signals has indicated that the major features of the fading can be summarized as follows:

1. Fading is more pronounced at sunrise than at sunset.

2. The sunrise NBA-Frankfurt path contains five amplitude minima; the NPM-Boulder path contains three minima.

3. The rate of phase change is approximately maximum at the times when signal is at minimum.

4. At each signal minimum there is a decrease in phase delay at sunrise and an increase at sunset.

5. At sunrise the depth of fading increases as the sunrise line approaches the transmitter.

6. Limited observations at two points along the same great-circle path from the transmitter indicate that fading occurs simultaneously and is of constant depth on the daylight side of the sunrise line.

The features listed above can be explained if it is assumed that under the nighttime ionosphere, first- and second-order waveguide modes are propagating. (These may be excited by the transmitting antenna.) It is then necessary to assume that at the sunrise boundary the nighttime first-order mode is converted into a daytime first-order mode. The second-order nighttime mode, on the other hand, is converted essentially into a first-order daytime mode. These two daytime modes cannot be subsequently separated since they have phase velocities and attenuation rates which are characteristic of the daytime ionosphere height and of the first-order mode.

Nighttime ionosphere changes phase and amplitude. In the nighttime ionosphere, however, the phase velocities and attenuation rates of the two modes are different. Thus the phase difference and relative amplitudes of the two modes depend on the distance of the SR line from the transmitter; as the SR line approaches the transmitter the two modes go in and out of phase at the boundary. Thus the phase of the first-order daytime mode (which is produced from the second-order nighttime mode) will also vary with this distance. An observer in daylight will observe the combination of the two modes; as the boundary moves, the phase and amplitude of the combination will alter. Therefore, the amplitude of the observed signal at all points east of the SR boundary will change from a maximum to a minimum as the boundary moves from a point at which the two first-order modes are in phase to a place where they are in antiphase.

At sunset, the transmitter is in daylight and excites essentially a first-order mode only. At the sunset boundary, this first-order daytime mode is converted into both a first- and a second-order mode, which again propagate and interfere in the nighttime portion of the path.

Careful application of this model indicates that it can account for all features observed and listed above. In addition, since the change in position of the sunrise line between the times of signal minima or maxima can be calculated, the difference in phase velocities of the two modes propagating in the nighttime waveguide can be calculated. From this result, using a suitable model of the nighttime ionosphere, it is possible to estimate the height of reflection.

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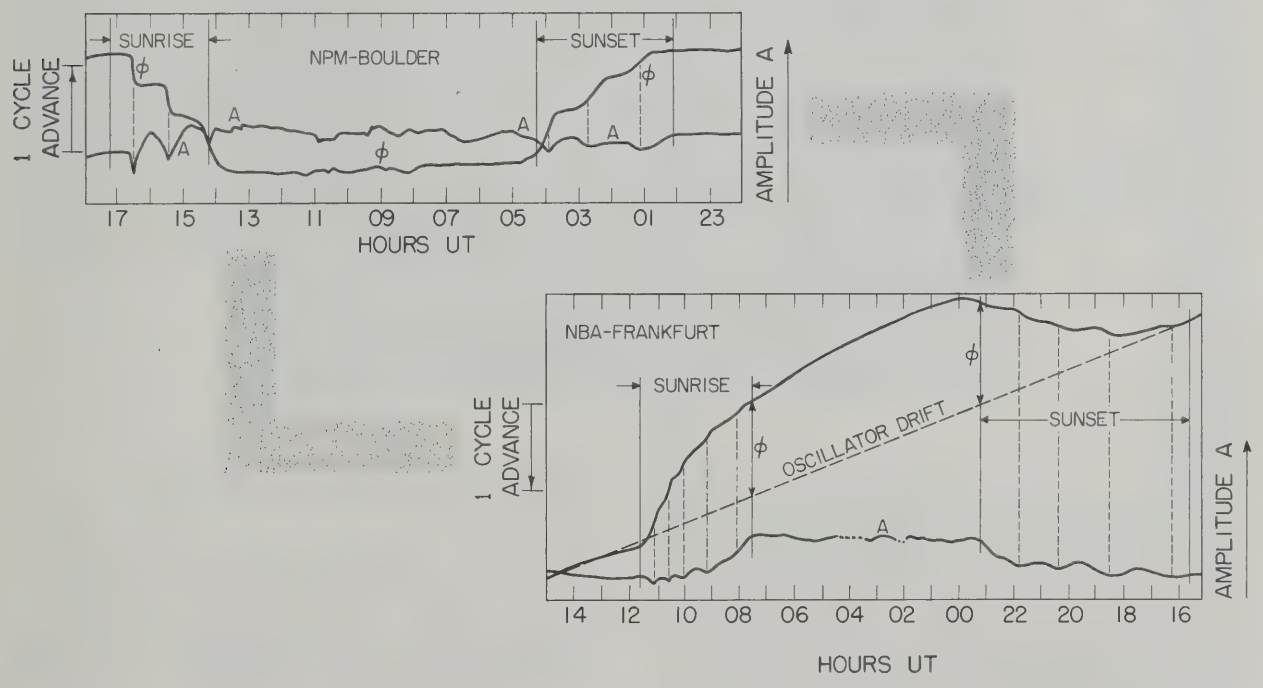
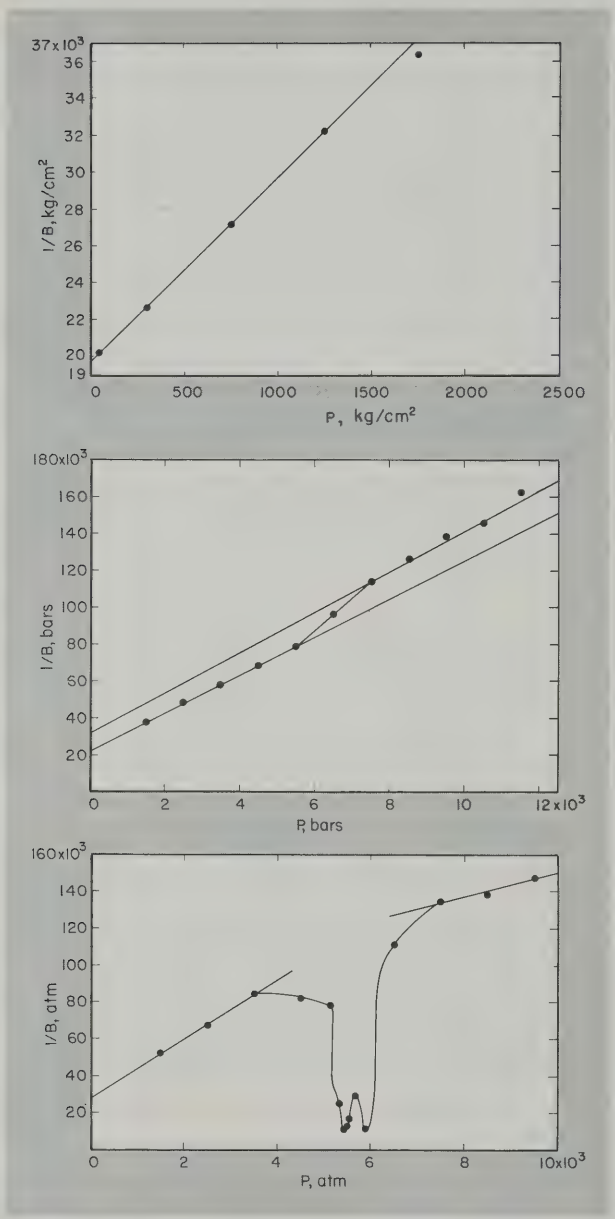
[1] Periodic fading of VLF signals received over long paths during sunrise and sunset, *by D. D. Crombie, J. Res. NBS* **68D** (*Radio Sci.*), *27–34* (*Jan. 1964*).



Typical diurnal phase (ϕ) and amplitude (A) variations of signals received at Boulder, Colo., from station NPM (Hawaii), and of those received at Frankfurt, Germany, from station NBA (Canal Zone) during January 1962.

Tait Equation Used in Polymer Studies

Study conducted by
L. A. Wood of the NBS
Institute for Materials Research



Various data were fitted to the Tait equation by plotting bulk modulus as a function of pressure. Naunton's data (top) *on a pure-gum vulcanizate of natural rubber give a linear plot; Adams and Gibson's data* (center) *on a natural rubber vulcanizate with 4 percent sulfur show a transition in the* 6.5×10^3 *bar* (6.5×10^8 *N/m²*) *region; and Weir's data* (bottom) *on tetrafluoroethylene also show a transition between 5000 and 6000 atm* (*5.065 to* 6.098×10^8 *N/m²*).

The 19th-century Tait equation for the pressure-volume relationship of sea water has been found useful in determining pressure-induced transitions in some polymers over a wide pressure range. A study [1] of the utility of the Tait equation for this purpose has recently been made at the Bureau.

The Tait equation is

$$\frac{V_0-V}{V_0}=c\log_{10}(1+P/b),$$

where V is volume, P is pressure, V_0 is volume when pressure is zero, and b and c are constants. The equation was first proposed in 1888 by P. G. Tait as the pressure-volume relation for sea water. Since then, the equation has had limited use in studies of some substances, such as liquid and crystalline hydrocarbons of low molecular weight, and recently, natural rubber. The equation in its original form has received little attention, probably because of the difficulty in determining appropriate values for the constants c and b.

For most polymers, the plot of volume (V) against pressure (P) is curved. Where pressure causes a transition in the polymer, the plot changes from a single continuous curve to two separate curves joined at the transition. The transition, however, is difficult to locate on the plot, as the junction of the curves is not readily apparent. Therefore, investigators have searched for an expression that would be linear both below and above the transition. Such an expression would permit the pressure-induced transition to be easily located, as a break between the two straight lines.

Dr. Wood took the first derivative of the Tait equation and obtained:

$$-\frac{\delta V}{\delta P}=(V_0)\,\frac{0.4343c}{P+b}.$$

Then, dividing both sides by V_0, he obtained

$$\frac{0.4343c}{P+b}=\frac{-1\delta V}{V_0\delta P}$$

which, by definition, is the compressibility (B).

When the values of B and P are known and the equation is put into the reciprocal form,

$$\frac{1}{B}=\left(\frac{1}{0.4343c}\right)P+\frac{b}{0.4343c},$$

the values for c and b are readily determined. A plot of the reciprocal of compressibility, bulk modulus $(1/B=K)$, against pressure (P) should be linear with an intercept $b/0.4343c$ and a slope $1/0.4343c$, if the Tait equation is valid.

A study using data of other investigators was initiated to determine the validity of the Tait equation over wide pressure ranges. The data used were from investigations of natural rubber,[2] a rubber vulcanizate with 4 percent sulfur,[3] and tetrafluorethylene.[4] For the pressure range from 0 to 2×10^3 kg/cm^2 (0 to 19.6×10^7 N/m^2), the Tait equation was found to be valid with values of c and b between 0.17 and 0.24, and 1000 and 4000 kg/cm^2 (9.8×10^7 and 39.2×10^7 N/m^2), respectively.

The resulting graphs of K versus P were linear in two parts with a definite break at the pressure-induced transitions. For the rubber vulcanizate with 4 percent sulfur, the transition occurred at approximately 6500 kg/cm^2 (63.7×10^7 N/m^2). The results of the study show that the Tait equation is valid over a wide range of pressures and that its use permits the rapid location and study of pressure-induced transitions in polymers.

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[1] *For further information, see* Utility of the Tait equation relating volume and pressure in the study of transitions in polymers, *by L. A. Wood, J. Polymer Sci. (Polymer Ltrs.Q* **2B,** *703–707 (July 1964).*

[2] *Ministry of Supply,* Rubber in Engineering (*W. J. S. Naunton, ed.), p. 30, Services Rubber Investigations, London, 1945.*

[3] L. H. Adams and R. E. Gibson, *J. Wash. Acad. Sci.* **20,** *213 (1930)*; *Rubber Chem. Technol.* **3,** *555 (1930).*

[4] *C. E. Weir, J. Res. NBS* **46,** *207 (1951) RP2192.*

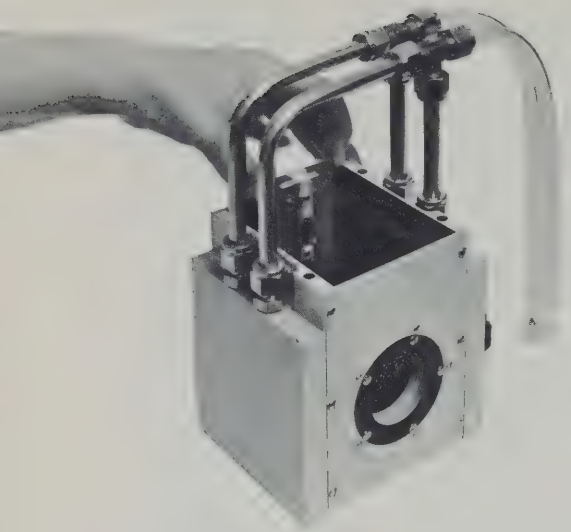
New JILA Laboratory Planned

The University of Colorado has announced a $900,000 grant from the National Science Foundation to pay approximately one-half the $2 million cost of constructing a new building to house the Joint Institute for Laboratory Astrophysics (JILA). The remaining money will come from a revolving fund provided to the University by the 1964 Colorado legislature to finance self-liquidating scientific buildings.

The Institute is a cooperative Center of NBS and the University for advanced research, study, and graduate education in astrophysics, atomic physics, aerodynamics, and other fields.

Plans for the structure have been completed by Harry Weese and Associates of Chicago, the architects. Completion of the building is scheduled for the spring of 1966.

JILA now is housed in the former State armory building just off the University of Colorado campus and in three ancient campus buildings. When completed, the JILA building will be part of a new multimillion dollar physics complex at the University.



Oxygen and Ozone

- *more durable asphalt*
- *reduced manufacturing costs*
- *less air pollution*
- *lower reaction time*

Experiments performed by P. G. Campbell and J. R. Wright of the NBS Institute for Applied Technology

Coating-grade asphalts are used for roofing on more than 80 percent of the residential buildings in the United States today. These asphalts are made commercially by blowing air at high flow rates through asphalt flux at high temperatures for many hours. By replacing the air with more reactive oxidants, such as oxygen and ozone, more durable asphalts can be produced in less time, according to a recent study by the NBS Institute for Applied Technology.[1]

Simple experimental apparatus, operating at temperatures lower than those employed commercially to produce coating-grade asphalts, was used. Asphalts produced in this way are more durable than commercially prepared asphalts because they are less susceptible to photo-oxidation effects, the major cause of chemical degradation in asphalts. As the reaction times required to produce these asphalts are considerably less than those required in the commercial method, the use of the more reactive oxidants promises to reduce manufacturing costs.

Conditions for Process

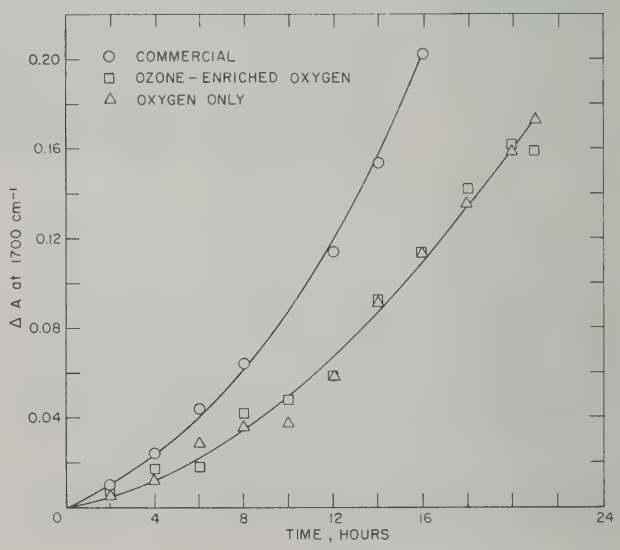
In the commercial process a batch of asphalt flux is heated to a high temperature (about 300 °C) and air is forced through the flux at high flow rates. This process takes from 4 to 40 hr, depending upon the type of flux used and the grade of asphalt desired. These conditions induce some thermal degradation of the asphalt, and cause a material loss of light oils, which can, if not controlled, contribute to air pollution.

Top of page: *Thin-film specimen of asphalt is placed in a controlled-temperature chamber which maintains specimen temperature at 0 °C during infrared analyses. When sealed, the chamber is placed in the spectrophotometer. Two potassium bromide windows permit infrared scanning of the specimen.*

In this study, Southeastern United States asphalt flux was used. However, additional experiments were conducted with asphalts from other sources, domestic and foreign, with similar results.

The experimental blowing apparatus consisted of a reaction vessel, hot plate, gas inlet tube with attached flowmeter, stirrer, and thermometers. In all experiments, the gas flow was maintained at 250 cm^3 per minute, the asphalt charge was 200 g, and the temperature was increased gradually from 110 to 158 °C during the hardening process. The air was replaced by ozone-enriched air, ozone-enriched oxygen, and pure oxygen. By operating at these lower temperatures, fewer light oils are driven off, reducing air pollution. Also, these temperatures are well below the asphalt flux flash point (about 300 °C).

Softening point determinations and infrared analysis were made periodically during the blowing process to monitor the reaction. Also, elemental analysis on the final product was performed.



Improve Asphalt Durability

Oxidation Effects

The relative effects of the gases on the softening point (ring and ball) of the asphalt flux were compared. The softening point of the unblown flux was 38 to 44 °C. After 24 hr reaction time with air, the softening point was 58 °C. Ozone-enriched air produced a softening point of 60 °C in approximately 12 hr and 82 °C after 24 hr. With oxygen, a softening point of 85 °C was obtained after 12 hr and one of 118 °C after 16.3 hr. Ozone-enriched oxygen increased the softening point at a slightly faster rate than oxygen, reaching 122 °C after 16 hr. Extrapolation of the ozone-enriched oxygen curve to a softening point of 58 °C indicates that slightly less than 4 hr would be needed to reach this softening point, which is approximately one-sixth the time required with air. Similarly, oxygen alone would require slightly more than 4 hr reaction time.

Softening Points

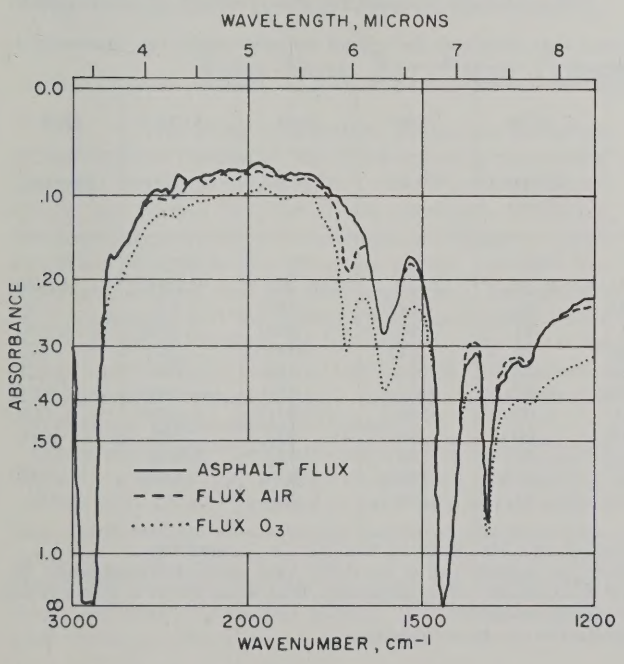
Infrared analysis was used to determine oxidation in the asphalts. Infrared analyses were made of unsupported films of the soft asphalt flux, asphalts of intermediate hardness, and the end products. As unhardened and partially hardened asphalt fluxes would melt in the infrared spectrophotometer, a controlled temperature chamber was used in the spectrophotometer to house the specimens and maintain specimen temperature at 0 °C. This chamber, equipped with two potassium bromide windows, permitted infrared scanning of the specimen.

The infrared spectrum for the air-blown flux showed the formation of an absorbance peak at about 1700 cm^{-1}, which was not present in the untreated asphalt flux. This absorbance is caused by carbonyl compounds, which form during oxidation. The carbonyl absorbance in the spectrum of the asphalt blown with ozone-enriched air showed a marked increase over that of the air-blown asphalt.

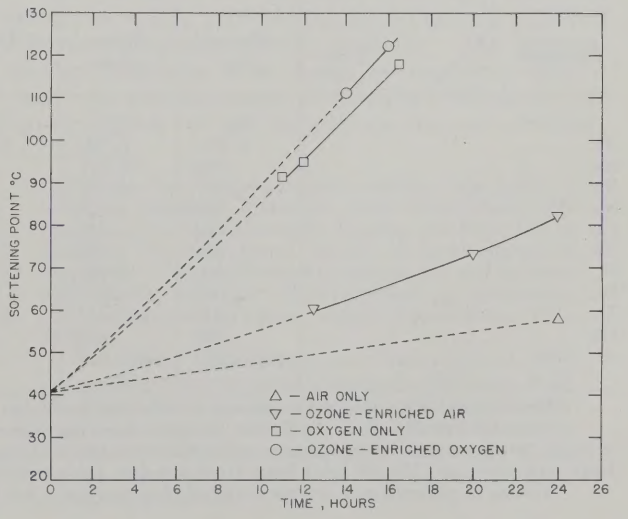
Photo-Oxidation Effects

Asphalt specimens blown with the various gases were exposed to carbon arc radiation in a weatherometer. This radiation causes photo-oxidation in the asphalt, which increases the carbonyl absorbance of the asphalt. Changes in absorbance were plotted against exposure time to determine the photo-oxidation rates, which are used to evaluate asphalt durability. The results show that the photo-oxidation rate of commercial air blown asphalt was greater than those of the other asphalts that had been hardened with more reactive oxidants.

[1] *For further information, see* Ozonation of asphalt flux, *by P. G. Campbell and J. R. Wright, Ind. and Eng. Chem. Prod. Res. and Dev.* **3**, *No. 3, 186–194 (Sept. 1964).*



Far left: *Relative effects of air, ozone-enriched, ozone-enriched oxygen, and pure oxygen on the softening point of southeastern U.S. asphalt flux.* Left: *Infrared spectra for southeastern U.S. flux, the air-oxidized flux, and the flux after reaction with ozone-enriched oxygen.* Below: *Results of photo-oxidation experiments. Carbonyl absorbance* (ΔA *at 1700* cm^{-1}) *is plotted against carbon exposure times at 49 °C and 40 percent relative humidity for three coating-grade asphalts. These asphalts were prepared from the same flux and all have softening points (S.P.) near 110–111 °C.*



Standard Materials

Trace Elements Certified in 13 Spectrometric Standards

"What is in this material?" . . . . "What are its properties?"

These questions are often asked by workers in industrial and research laboratories. The Bureau helps them to find answers by issuing samples of Standard Reference Materials [1] which are used to calibrate analytical equipment or measurement systems. These materials may be certified for chemical composition or for some important physical property.

In standard samples of alloys, it generally has been necessary in the past to certify only the main (or specified) constituents. However, as minute amounts of other elements may have an effect on the alloys' properties, it is desirable and often imperative to know which other elements are present and in what amount. These trace elements may be derived from the raw materials, picked up from furnace linings, or added deliberately to enhance performance.

At the Bureau, trace elements are purposely added to some standard reference materials to provide a useful

(*continued opposite*)

TABLE 1. *Trace elements added to certificate of analyses* of cartridge brass standards for optical emission and x-ray spectroscopic analysis*

Elements \ NBS No. [1]	[2] C1100	1101 C1101	1102 C1102
	%	%	%
Aluminum	0.008	0.0006	0.0007
Antimony	.018	.012	.005
Arsenic	.019	.009	.004
Beryllium	$.001_5$	$.0005_5$	$.0000_3$
Bismuth	.0010	$.000_4$	$.000_5$
Cadmium	.013	$.005_5$	$.004_5$
Silver	.019	.003	.0010
Tellurium	$.003_5$	$.001_5$	$.000_3$

*The original Provisional Certificate of Analyses provided values for Cu, Zn, Pb, Fe, Sn, Ni, Mn, P, and Si.

[1] Size and metallurgical condition: 1100 series are wrought samples 1¼ in. in diameter, ¾ in. thick. C1100 series are chill-cast samples 1¼ in. square, ¾ in. thick.

[2] The wrought material for this standard is not available.

TABLE 2. *Trace elements added to certificate of analyses* of white-cast iron standards for optical emission and x-ray spectroscopic analyses*

Element \ Type / NBS No.[1]	1176	1177	1178	1179	1180	1181	1182	1183
	Piston ring	Wear plate	Die	Brake drum	Mold	Special 1	Special 2	Special 3
	%	%	%	%	%	%	%	%
Ti	0.20	0.08_0	0.17	0.03_0	0.53	(0.04)	0.034	0.009
As	.008	(0.0_1)	.024	$(.0_5)$	.060	.067	(.15)	.17
Sb	[2] (.3)	(.1+)	.11	.11	.063	.005	(.004)	(.005)
Sn	.006	(.02)	.086	.12	$.02_5$	.041	.016	$.15_5$
Co	.006	$.10_5$	.060	.031	$.03_5$	.012	.004	.017
Te	$(.01_4)$	$(.01_4)$	$(.004)$	$(.02_4)$	$(.02_4)$	$(.02_5)$	$(.009)$	$(.02_2)$
B	$(.00_1)$	$(.02_5)$	$(.1_1)$	(.05)	$(.00_2)$	$(.001_4)$	(.009)	(.006)
Bi	.007	.017	.013	$.004_5$	$.002_5$	$.002_7$	.006	.016
Zr	(<.01)	(<.01)	$(.01_4)$	(<.01)	(<.01)	$(.02_5)$	$(.01_0)$	$.12_4$
Pb	.002	.002	$.00_4$	.013	$.004_3$	(.008)	$.004_6$	$.005_5$
Al	(<.01)	(<.01)	$(.01_6)$	(<.01)	$(.04_0)$	$(.01_5)$	(<.01)	$.01_7$

*The original Provisional Certificate of Analyses provided values for C, Mn, P, S, Si, Cu, Ni, Cr, V, and Mo.

[1] Size and metallurgical condition: Samples are approximately 1¼ in. square and ¾ in. thick; they were chill-cast white by a rapid unidirectional solidification technique and the addition of innoculants, when necessary, and were given a stress relief heat treatment at 1100 °F for 1 hour (this is below the graphitizing temperature).

[2] Values in parentheses are *not* certified, but are given for information on the composition.

Stratton Award Recipients

Ernest Ambler, Raymond Hayward, Ralph Hudson, and Dale Hoppes received the 1964 Samuel Wesley Stratton award for their work in demonstrating that the quantum mechanics law of parity conservation does not hold in weak interactions. The award, named for the Bureau's first director, was first presented in 1962. It recognizes outstanding scientific or engineering achievements by members of the Bureau staff in support of the NBS mission. Each recipient receives a bronze plaque and shares equally in a $5,000 award.

Standard Materials—Cont.

concentration range—as an example, for spectrometric analysis at the parts-per-million level. Recently, to increase the value of 13 spectrometric standard samples to industry, the NBS Office of Standard Reference Materials has completed the certification of a number of trace elements; their values have now been added to the previously available certification of analyses covering the main constituents.

The 13 standards involved were 5 NBS Cartridge Brass Spectrometric Standards and 8 NBS White Cast Iron Spectrometric Standards.[2] Two or more completely independent methods of analysis were employed for the determination of each newly certified element. These elements are usually determined and certified in cooperative programs with industrial laboratories. The certification of the trace elements in the brass standards, however, was carried out at NBS without outside cooperation. This marks the first time that such a program was performed entirely by the Bureau. The compositions of the brass standards are shown in table 1, and those of the cast iron standards in table 2.

Two New Beryllium Copper Standards to be Prepared

The Office of Standard Reference Materials of the National Bureau of Standards recently announced plans for the preparation of two new beryllium-copper alloys, No. 10 and No. 25. These standards are being developed to meet the critical needs of industry, where analytical standards for these alloys are required for both production control and consumer acceptance. With numerous and increasing specialized applications of the alloys, the tolerances for variations in behavior and properties have become severe, and the proposed NBS standards are needed to maintain the chemical composition within allowable limits.

These alloys are applied broadly in the electrical and electronics industries, in instrumentation, in welding, and in safety tools. Because of their unique properties, they are found in almost every military, space, oceanographic, and aviation system. It is estimated that about $50 million per year is spent for the material itself, and many times that amount for the actual hardware items produced.

Once prepared and analyzed, these new analytical standards will be added to the more than 400 standard reference materials already available. The preparation and analytical work should be completed within a year. An announcement of these two standards will appear in this column as soon as they are available.

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[1] *Standard reference materials available from NBS are listed in* Standard Materials, NBS Misc. Publ. 260, Standard Reference Materials: Catalog and Price List of Standard Materials Issued by the National Bureau of Standards. *This publication is in press and will be available shortly from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402.*

[2] *For further information, see* Preparation of NBS Copper Base Spectrochemical Standards, NBS Misc. Publ. 260–2, *and* Preparation of NBS White Cast Iron Spectrochemical Standards, NBS Misc. Publ. 260–1; *both may be obtained from the Superintendent of Documents, Washington, D.C., 20402. Misc. Publ. 260–1, 30 cents; Misc. Publ. 260–2, 35 cents.*

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CRPL Ionospheric Predictions for April 1965. Three months in advance. Number 25, issued January 1965. 15 cents. Annual subscription: $2.50; 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year. Annual subscription: Domestic, $4; foreign, $4.75. Single copy, 70 cents.

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Section C. Engineering and Instrumentation. Issued quarterly. Annual subscription: Domestic, $2.25; foreign, $2.75. Single copy, 75 cents.

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Current Issues of the Journal of Research

J. Res. NBS **69B** *(Math. and Math. Phys.), No. 1 (Jan–Mar. 1965).*

Lectures on matroids. W. T. Tutte.
Menger's theorem for matroids. W. T. Tutte.
Single-element extensions of matroids. H. H. Crapo.
Minimum partition of a matroid into independent subsets. J. Edmonds.
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On measurable sets and functions. A. J. Goldman.
Characteristic formulation for nonperiodic solutions of Mathieu's equation. T. Tamir and H. C. Wang.
On the surface duality of linear graphs. J. Edmonds.
Maximum matching and a polyhedron with 0,1-vertices. J. Edmonds.

Radio Sci. J. Res. NBS **69D**, *No. 2 (Feb. 1965).*

Electromagnetic wave penetration of reentry plasma sheaths. M. P. Bachynski.
On the use of refractive index diagrams for source-excited anisotropic regions. L. B. Felsen.
Surface waves along a perfectly conducting plane covered with semi-infinite magneto-plasma. S. Adachi and Y. Mushiake.
On the point of emergence of a microwave beam entering a linearly graded plasma. A. L. Cullen.
Self and mutual admittances of waveguides radiating into plasma layers. J. Galejs.
Effect of electron collisions on the formulas of magneto-ionic theory. K. G. Budden.
Momentum transfer collisions in oxygen for thermal electrons. M. H. Mentzoni.
Experimental studies of perturbations in ionospheric plasma. L. H. Heisler.
Electromagnetic scattering by gyrotropic cylinders with axial magnetic fields. W. C. Y. Lee, L. Peters, Jr., and C. H. Walter.
Radar cross sections of plasma bodies at the plasma frequency. L. Peters, Jr.
Electroacoustic waves excited by a space vehicle in ionized atmosphere and its effect on radar return. K.-M. Chen.
Discussion on basic equations with source terms in compressive plasmas. K.-M. Chen.
Scattering of electromagnetic and electroacoustic waves by a cylindrical object in a compressible plasma. J. R. Wait.
Multiphase periodic very-low-frequency emissions. N. Brice.
Radio studies of the high-latitude ionosphere during the solar eclipse of 20 July 1963. R. D. Hunsucker.
VLF and LF fields propagating near and into a rough sea. R. M. Lerner and J. Max.
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